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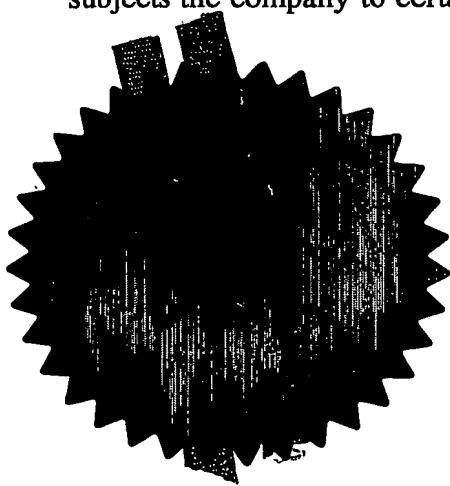
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4. Title of the invention

LEAN NOx CATALYST

5. Name of your agent (if you have one)

ANDREW DOMINIC NUNN

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Claim(s)

Abstract

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Drawing(s)

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LEAN NO_x CATALYST

5 This invention relates to a lean NO_x catalyst (LNC) comprising a support comprising silver.

10 LNCs are sometimes also referred to in the literature as non-selective catalytic reduction (NSCR) catalysts, hydrocarbon selective catalytic reduction (HC-SCR) catalysts, lean NO_x reduction catalysts, "DeNO_x catalysts" and NO_x occluding catalysts.

15 In lean NO_x catalysis, hydrocarbons (HC) react with nitrogen oxides (NO_x), rather than oxygen (O₂), to form nitrogen (N₂), carbon dioxide (CO₂) and water (H₂O) according to equation (1):



20 The competitive, non-selective reaction with oxygen is given by Equation (2):



25 There are two preferred groups of LNC to selectively promote the desired reaction (1) described in the literature: platinum (Pt) on alumina (Al₂O₃) and copper (Cu)-substituted zeolite such as Cu/ZSM-5.

30 Pt-based catalysts tend to operate at relatively low temperature (peak activity ~250°C) and have a relatively narrow temperature window for lean NO_x activity. Another problem is that the popular Pt/Al₂O₃ favours the formation of N₂O over N₂ at relatively low temperatures.

35 Zeolite-based LNCs have a wider temperature window than Pt-based LNCs and also operate at higher temperatures (peak activity ~400°C). However, a problem associated with zeolite-based LNCs such as Cu/ZSM-5 is that they can become

deactivated in use through lean hydrothermal ageing. This is caused by sintering of the Cu component and/or de-alumination of the zeolite.

Another known LNC is silver (Ag) supported on Al_2O_3 as described in EP 5 0658368.

We have investigated such silver-based lean NO_x catalysts and we have determined that their activity at lower temperatures is suppressed by coke formation, which may be one reason why it has not found wider acceptance in the industry. We 10 have now discovered that by combining a known silver-based lean NO_x catalyst with a partial oxidation catalyst (POC) that it is possible to suppress or avoid low temperature coke formation to provide an improved silver-based catalyst for low temperature NO_x conversion.

15 A partial oxidation catalyst (POC) is a catalyst that promotes the partial oxidation of hydrocarbons in exhaust gas of a lean-burn internal combustion engine to carbon monoxide (CO), hydrogen gas (H_2) and partially oxygenated hydrocarbon species.

EP 1008379 describes a method and device for treating NO_x in an internal 20 combustion engine exhaust system comprising a POC upstream of a NO_x trap, wherein hydrocarbons introduced into the exhaust system upstream of the POC are converted to CO and H_2 for regenerating the NO_x trap.

According to a first aspect, the invention provides a lean NO_x catalyst (LNC) 25 composition comprising silver on at least one support and at least one (POC).

An advantage of the present invention is that low temperature NO_x conversion activity e.g. from 200-350°C is enhanced compared with known $\text{Ag}/\text{Al}_2\text{O}_3$ catalysts. By combining the lean NO_x catalyst composition of the present invention with lean NO_x 30 catalysts with activity at higher temperatures, e.g. the known $\text{Ag}/\text{Al}_2\text{O}_3$ lean NO_x catalysts or the above-mentioned Cu/ZSM5, it should be possible to obtain an exhaust system having a wide temperature window for lean NO_x activity.

In one embodiment, the at least one partial oxidation catalyst comprises manganese (Mn), iron (Fe) cerium (Ce), nickel (Ni), rhodium (Rh) or praseodymium (Pr).

5 According to a further embodiment, the LNC composition and the at least one POC according to the invention comprises at least stabiliser selected from zirconium (Zr), lanthanum (La), alumina (Al_2O_3), yttrium (Y), Pr and neodymium (Nd). Where present, the at least one stabiliser can be present in the weight ratio of from 2:98M to 50:50M, where M is the at least one of Mn, Fe, Ce, Ni, Rh and Pr.

10 According to a further embodiment, the at least one POC comprises an oxide, a compound oxide or a mixed oxide of the at least one of Mn, Fe, Ce, Ni, Rh or Pr. In a particular embodiment, the at least one POC is bulk CeO_2 , a mixed oxide or compound oxide of CeO_2 and ZrO_2 or a mixed oxide or compound oxide of a manganese oxide and
15 ZrO_2 .

20 In embodiments according to the invention, the support for use in the invention can comprise alumina, ceria, zirconia, titania, silica, magnesia, chromia, or a mixture, composite oxide or mixed oxide of any two or more thereof. Illustrative embodiments of such composite oxides or mixed oxides include a non-zeolite silica-alumina, a silica-zirconia, alumina-zirconia, alumina-chromia, alumina-ceria, ceria-titania or ceria-zirconia.

25 The at least one support and/or the at least one POC can be doped with at least one of lanthanum, barium, cerium, tungsten, silica and manganese to improve the stability of the support to high temperature ageing, act as a lean NO_x promoter and/or improve the oxygen storage capacity of the system, the latter particularly in the case of manganese.

30 In embodiments, the silver in LNC compositions according to the invention can be present in the range 0.5 to 10 wt% based on the total weight of the at least one support.

In further embodiments comprising a mixture of the silver on the at least one support and the at least one POC, the weight ratio of the at least one support to the at least one particulate oxide material in the mixture can be from 20:1 to 1:5, such as from 10:1 to 1:1.

5

Methods of preparing the LNC compositions according to the invention are known and include wet impregnation, ion-exchange and co-precipitation. A detailed discussion of which methods shall not be made here, as the skilled person is aware of such techniques.

10

According to a second aspect, the invention provides an exhaust system for a lean-burn internal combustion engine comprising a LNC composition according to the invention.

15

In one embodiment the exhaust system can comprise means for introducing a reductant into an exhaust gas upstream of the LNC composition. Such means can comprise at least one of: means for injecting the reductant into the exhaust gas; means for adjusting the ignition timing of at least one engine cylinder; and means for adjusting the engine air-to-fuel ratio.

20

In a particular embodiment, the reductant for use in the exhaust system according to the invention comprises a hydrocarbon, particularly a hydrocarbon fuel that powers the engine.

25

Means for controlling the reductant introducing means may also comprise part of the exhaust system and particularly can comprise a pre-programmed electronic control unit.

30

According to a particular aspect, the invention the means for introducing reductant is arranged so that reductant is introduced when the LNC composition is at 200-350°C.

The exhaust system according to the invention can take a number of forms. In one embodiment, the POC component comprises the silver support, e.g. bulk CeO₂, and the component is applied e.g. as a washcoat to a suitable substrate. In a second embodiment, the POC component and the silver component are on separate supports and 5 a mixed washcoat is applied on the substrate. In a third embodiment, the two components are applied in separate layers to a substrate, and, in a particular embodiment, the POC component is in an upper layer over the silver component. Of course, the silver component can be layered on the POC component, if desired.

10 In an alternative arrangement, however, the POC component and the silver component are segregated by coating each component on a separate zone of a substrate i.e. with no overlaying. For example, half of a substrate can be coated with the silver component and the other half with the POC component.

15 According to a further alternative embodiment, each component is coated on a separate substrate brick.

In either of the latter two embodiments, desirably the POC component is upstream of the silver component. This is so that coking on the silver component can be 20 reduced or prevented because the POC component can partially oxidise the hydrocarbons. For the same reason, the embodiment described above wherein one component is layered over the other, the embodiment wherein the POC is layered over the silver component is preferred.

25 However, if desired, a means for introducing HC into the exhaust gas between the substrates can be provided in the embodiment wherein the silver component and the POC component are carried on separate substrates.

A suitable substrate for use in the present invention can be a flow-through 30 monolith of metal or ceramic construction. Where ceramic, the substrate can be cordierite or silicon carbide, for example.

According to a third aspect, the invention provides a lean-burn internal combustion engine including an exhaust system according to the invention. The engine can be, for example, a diesel engine, preferably a heavy-duty diesel engine.

5 According to fourth aspect, the invention provides a method of reducing NO_x in an exhaust gas of a lean burn internal combustion engine to N₂ using a lean NO_x catalyst (LNC) comprising silver on at least one support and at least one partial oxidation catalyst, which process comprising contacting the LNC with the exhaust gas comprising a reductant, optionally at from 200-350°C.

10 In order that the invention may be more fully understood, the following Examples are provided by way of illustration only and with reference to the accompanying drawings, in which:

15 Figure 1 is a graph comparing %NO_x conversion between steady state and transient modes for Comparative Example 1 as a function of temperature;

20 Figure 2 is a graph showing the NO_x activity over the Catalyst of Example 1 compared with the Catalyst of Comparative Example 2 in the steady state mode as a function of temperature;

Figure 3 is a graph showing the outlet NO_x concentration as a function of time at 200°C, 250°C and 300°C over 2%Ag/Al₂O₃ and the 2Ag/Al₂O₃-CeO₂ mixture in the steady mode;

25 Figure 4 is a graph showing is a graph showing the effect of ramp down (steady state 15 minutes) and ramp up (steady state 15 minutes) on NO_x conversion of 2Ag/Al₂O₃-CeO₂ (4:1);

30 Figure 5 is a graph showing the effect of ramp down (steady state 15 minutes) and ramp up (steady state 15 minutes) on HC conversion of 2Ag/Al₂O₃-CeO₂ (4:1); and

Figure 6 is a graph showing the effect of ageing on 2Ag/Al₂O₃ and 2Ag/Al₂O₃-CeO₂ on NO_x conversion.

Comparative Example 1

Catalyst Preparation

5

An impregnated Ag catalyst at 2wt% Ag based on the weight of the Al₂O₃ support (referred to hereinafter as 2Ag/Al₂O₃) was made using silver nitrate by a wet impregnation method. The silver nitrate was dissolved in the appropriate amount of water measured from the pore volume of an Al₂O₃ support. The solution was then added to the Al₂O₃ with mixing. After drying overnight, the sample was calcined in air at 10 500°C for 2 hours. The resulting catalyst was pelletised and particles in the size range 250< d <355 µm were selected.

10

15

Comparative Example 2

Catalyst Preparation

The pelletised catalyst particles in the size range 250< d <355 µm prepared according to Comparative Example 1 were physically mixed with pelletised particles of cordierite of the same size in a weight ratio of 4:1 2Ag/Al₂O₃:cordierite.

20

Example 1

Catalyst Preparation

25

The pelletised catalyst particles in the size range 250< d <355 µm prepared according to Comparative Example 1 were physically mixed with pelletised particles of bulk ceria of the same size in a weight ratio of 4:1 2Ag/Al₂O₃:ceria.

30

Example 2

Activity Measurements

The activity of catalysts to reduce NO to N₂ using MK1 (diesel fuel) as the reductant was measured in a simulated catalyst activity test (SCAT) gas rig species. The simulated exhaust gas composition used in the test was as follows: NO 500ppm,

C1 2250ppm (MK1 diesel fuel), CO 200ppm, O₂ 9%, H₂O 4.5%, CO₂ 4.5%, N₂ balance (C1:NO_x 4.5:1). The data was collected in one of two modes:

5 (i) steady state conditions (with 15 minutes dwell at each temperature) or transient ramp up at 10°C per minute temperature from 200°C to 500°C; and

10 (ii) transient ramp up at 10°C per minute temperature from 200°C to 500°C.

Comparative Example 3

Results of Activity Measurements on Catalyst of Comparative Example 1

15 Figure 1 shows a graph comparing %NO_x conversion between steady state and transient modes for the catalyst of Comparative Example 1 as a function of temperature. It can be seen that the sample is very active for NO_x conversion between 300°C and 550°C (~60% conversion) in the transient mode, but it is severely deactivated in the steady state mode compared with the transient mode. We believe that this deactivation is caused by carbon species ("coke") deposited on the catalyst surface after exposure to the 20 gas mixture for long periods at low temperature. Such conditions can be encountered in the exhaust of a diesel engine. Reasons for the above conclusion include our finding that catalyst deactivation is less severe at a C1:NO_x ratio of 3:1 and activity is restored by heating the deactivated catalyst in the reaction mixture at 550°C (results not shown).

25

Example 2

Results of Activity Measurements on Catalyst of Example 1

30 Figure 2 shows the %NO_x conversion activity over the 2Ag/Al₂O₃-CeO₂ catalyst of Example 1 compared with the 2Ag/Al₂O₃ catalyst of Comparative Example 2 in the steady state mode as a function of temperature. The results show that the addition of particulate CeO₂ to the 2Ag/Al₂O₃ catalyst greatly enhances its activity between 200°C-350°C. The control sample mixed with cordierite has no significant activity in this temperature region. However, the high temperature activity is lower with the

2Ag/Al₂O₃-CeO₂ mixture due to the non-selective HC oxidation over the CeO₂. A combination of these catalysts components (2Ag/Al₂O₃ and 2Ag/Al₂O₃-CeO₂) may provide a wider activity window.

5 Figure 3 shows the outlet NO_x concentration (ppm) as a function of time at 200°C, 250°C and 300°C over a 2Ag/Al₂O₃ catalyst of Comparative Example 1 and the 2Ag/Al₂O₃-CeO₂ catalyst of Example 1 in the steady mode. The conversion over the simulated exhaust gas composition is higher compared with the 2Ag/Al₂O₃ only sample. The 2Ag/Al₂O₃-CeO₂ catalyst has a lower decay in activity with time, especially at 10 250°C and above than the 2Ag/Al₂O₃ catalyst. We believe that this is due to the absence of any coke deposition on the 2Ag/Al₂O₃-CeO₂ catalyst.

Example 3

Results of Activity Measurements: Steady State Ramp-Up and Ramp-Down on

Catalyst of Example 1

15 The activity of the 2Ag/Al₂O₃-CeO₂ catalyst under steady state conditions from 500°C to 200°C and then from 200°C to 500°C (C1:NO_x=4.5:1) was measured and the results are shown in Figures 4 and 5 respectively. The NO_x and HC conversion are similar between ramp down and ramp up and no catalyst deactivation was observed. 20 The effect of lowering the C1:NO_x ratio to 3:1 is also shown. In contrast to previous results with 2Ag/Al₂O₃ alone, the NO_x conversion is lower at low temperature at a C1:NO_x ratio of 3:1. The results are summarised in Table 4.

Table 1: Effect of CeO₂ addition on 2Ag/Al₂O₃ lean NO_x activity.

Catalyst	NO _x conversion			HC conversion		
	200°C	250°C	300°C	200°C	250°C	300°C
2Ag/Al ₂ O ₃ +corderite	8	7	13	--	--	--
2Ag/Al ₂ O ₃ - CeO ₂	12	41	37	1	43	56
2Ag/Al ₂ O ₃ - CeO ₂ *	14	24	28	19	56	68

25 *C1:NO_x=3:1

Example 4
Catalyst Ageing

5 The 2Ag/Al₂O₃ and 2Ag/Al₂O₃-CeO₂ catalysts were lean hydrothermally aged in a 10% H₂O and air mixture at 700°C for 16 hours and the steady state activity of the resulting catalysts are shown in Fig 6. It can be seen that even after the high temperature ageing, the presence of CeO₂ is beneficial to the low temperature activity.

CLAIMS:

1. A lean NO_x catalyst (LNC) composition comprising silver on at least one support and at least one partial oxidation catalyst (POC).

5

2. A LNC composition according to claim 1, wherein the at least one POC comprises manganese (Mn), rhodium (Rh), iron (Fe) cerium (Ce), nickel (Ni) or praseodymium (Pr).

10 3. A LNC composition according to claim 2, wherein the at least one POC comprises at least stabiliser selected from zirconium (Zr), lanthanum (La), alumina (Al₂O₃), yttrium (Y), Pr and neodymium (Nd).

15 4. A LNC composition according to claim 3, wherein the at least one stabiliser is present in the weight ratio 2:98M to 50:50M, where M is the at least one of Mn, Rh, Fe, Ce, Ni and Pr.

20 5. A LNC composition according to claim 2, 3 or 4, wherein the at least one POC comprises an oxide, a compound oxide or a mixed oxide of the at least one of Mn, Rh, Fe, Ce, Ni or Pr.

25 6. A LNC composition according to any of claims 2 to 5, wherein the at least one POC is bulk CeO₂, a mixed oxide or compound oxide of CeO₂ and ZrO₂ or a mixed oxide or compound oxide of a manganese oxide and ZrO₂.

25

7. A LNC composition according to any preceding claim, wherein the at least one support comprises alumina, ceria, zirconia, titania, silica, magnesia, chromia, or a mixture, composite oxide or mixed oxide of any two or more thereof.

30 8. A LNC composition according to claim 7, wherein the composite oxide or mixed oxide comprises a non-zeolite silica-alumina, a silica-zirconia, alumina-zirconia, alumina-chromia, alumina-ceria, ceria-titania or ceria-zirconia.

9. A LNC composition according to claim 7 or 8, wherein the at least one support and/or the at least one POC is doped with at least one of lanthanum, barium, cerium, tungsten, silica and manganese.

5 10. A LNC composition according to any preceding claim, wherein the or each silver support comprises the POC.

11. A LNC composition according to any preceding claim, wherein the silver is present in the range 0.5 to 10 wt% based on the total weight of the at least one support.

10 12. A LNC composition according to any preceding claim comprising a mixture of the silver on the at least one support and the at least one POC, wherein the weight ratio of the at least one support to the at least one particulate oxide material in the mixture is from 20:1 to 1:5, such as from 10:1 to 1:1.

15 13. A substrate comprising a first layer of the or each supported silver component and a second layer over the first layer comprising the or each POC.

14. An exhaust system for a lean-burn internal combustion engine comprising a LNC composition according to any preceding claim.

20 15. An exhaust system according to claim 14, comprising a substrate comprising the or each POC component on an upstream zone and the or each supported silver component on a downstream zone.

25 16. An exhaust system according to claim 14 or 15, comprising means for introducing a reductant into an exhaust gas upstream of the LNC composition.

30 17. An exhaust system according to claim 16, wherein the reductant introducing means comprises at least one of: means for injecting the reductant into the exhaust gas; means for adjusting the ignition timing of at least one engine cylinder; and means for adjusting the engine air-to-fuel ratio.

18. An exhaust system according to claim 14, 15, 16 or 17 wherein the reductant is a hydrocarbon.

19. An exhaust system according to claim 18, wherein the hydrocarbon is the fuel
5 that powers the engine.

20. An exhaust system according to any of claims 14 to 19, comprising means for controlling the reductant introducing means.

10 21. An exhaust system according to claim 20, wherein the control means introduces reductant when the LNC composition is at 200-350°C.

22. An exhaust system according to claim 20 or 21, wherein the control means includes a pre-programmed electronic control unit.

15 23. A lean-burn internal combustion engine including an exhaust system according to any of claims 14 to 22.

24. An engine according to claim 23, wherein it is a diesel engine, preferably a
20 heavy-duty diesel engine.

25. A method of reducing NO_x in an exhaust gas of a lean burn internal combustion engine to N₂ using a lean NO_x catalyst (LNC) comprising silver on at least one support and at least one partial oxidation catalyst (POC), which process comprising contacting the LNC with the exhaust gas comprising a reductant, optionally at from 200-350°C.

26. A lean NO_x catalyst composition substantially as described herein.

27. An exhaust system for a lean-burn internal combustion engine substantially as
30 described herein.

28. A lean burn internal combustion engine substantially as described herein.

29. A method of reducing NO_x in an exhaust gas of a lean burn internal combustion engine to N₂ using a lean NO_x catalyst substantially as described herein.
30. A substrate substantially as described herein.

5

LEAN NO_x CATALYST

Abstract

10 A lean NO_x catalyst (LNC) composition comprising silver on at least one support and at least one partial oxidation catalyst (POC).

[No Figure]

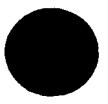
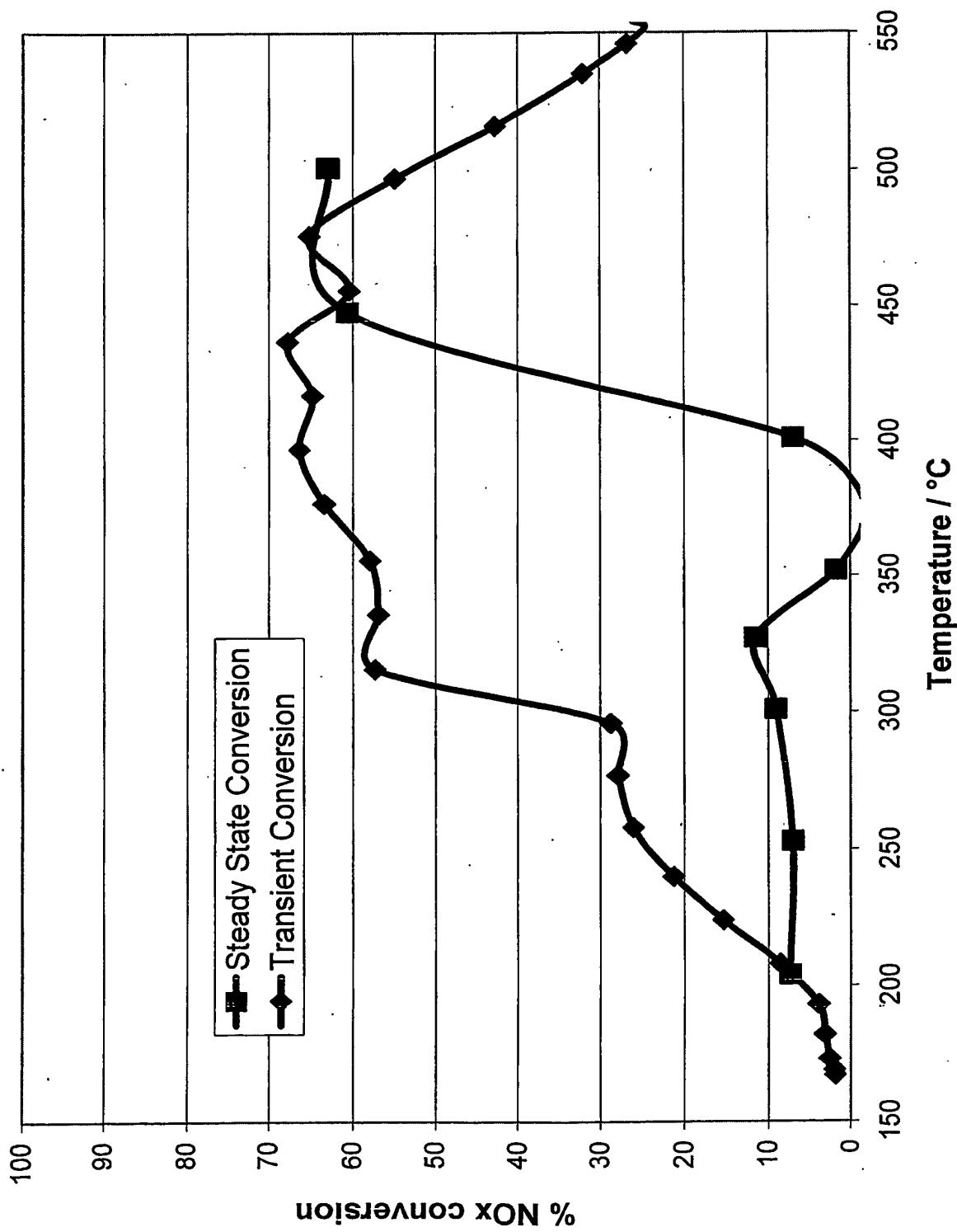


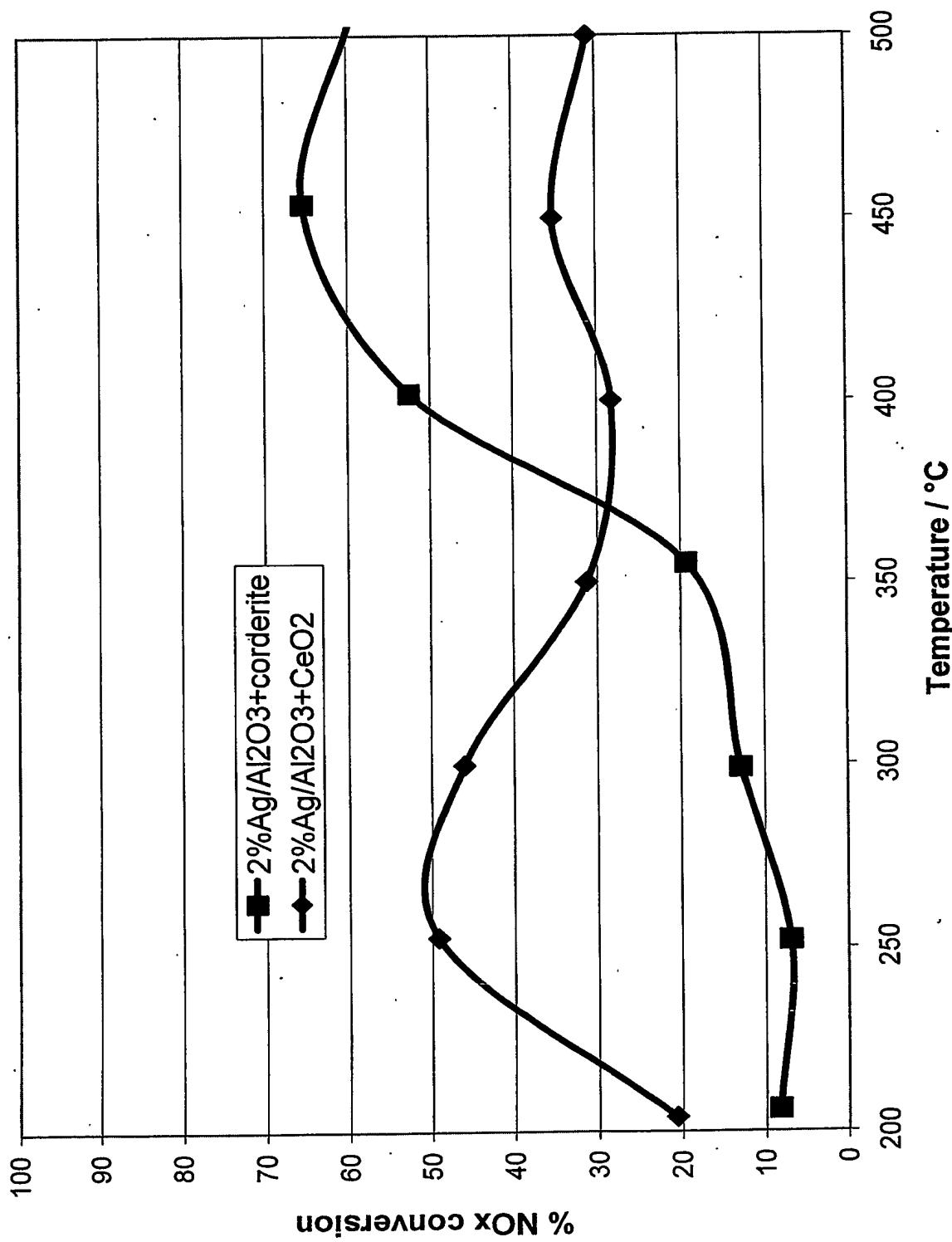
FIGURE 1





2/6

Figure 2



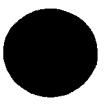


Figure 3

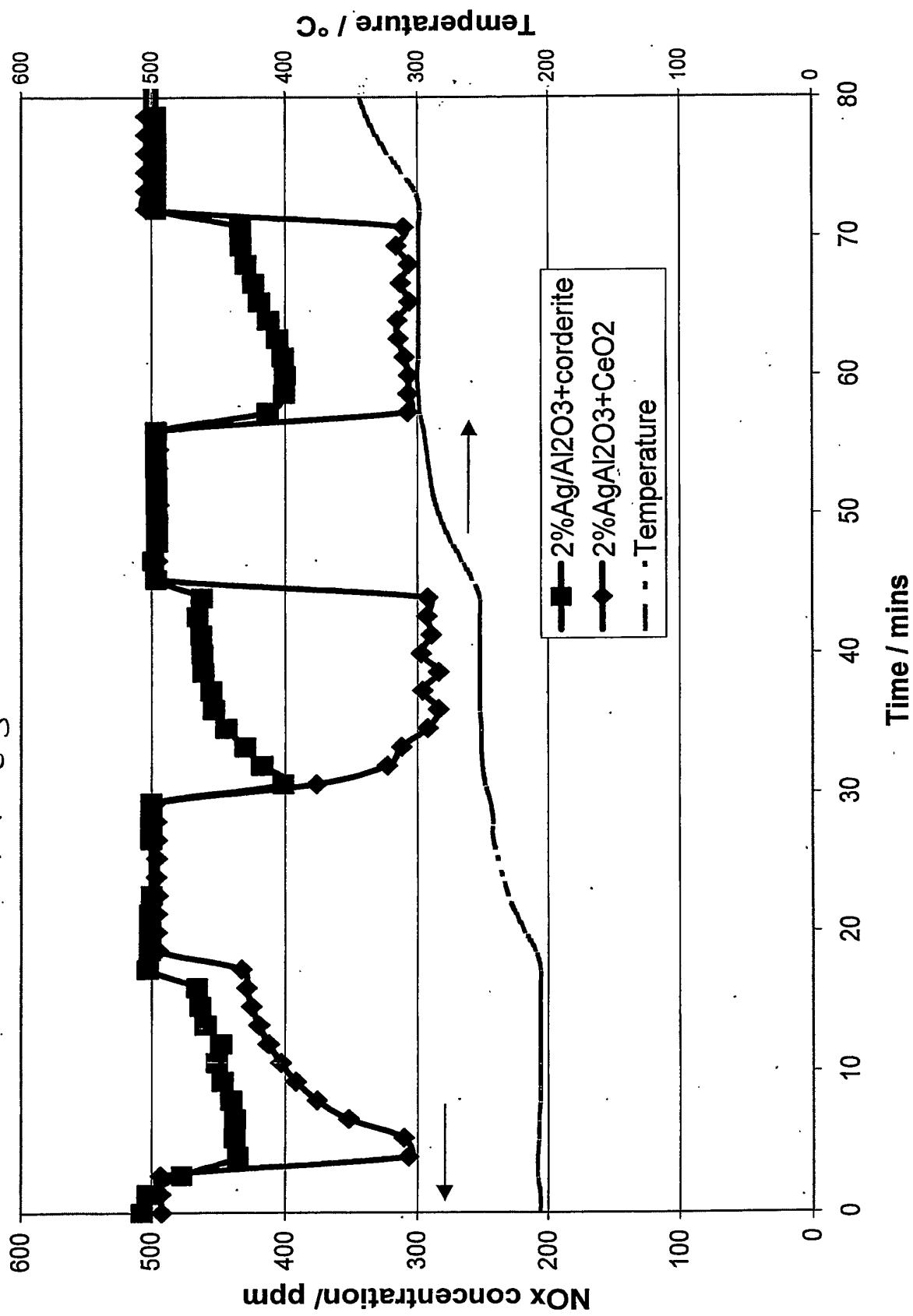
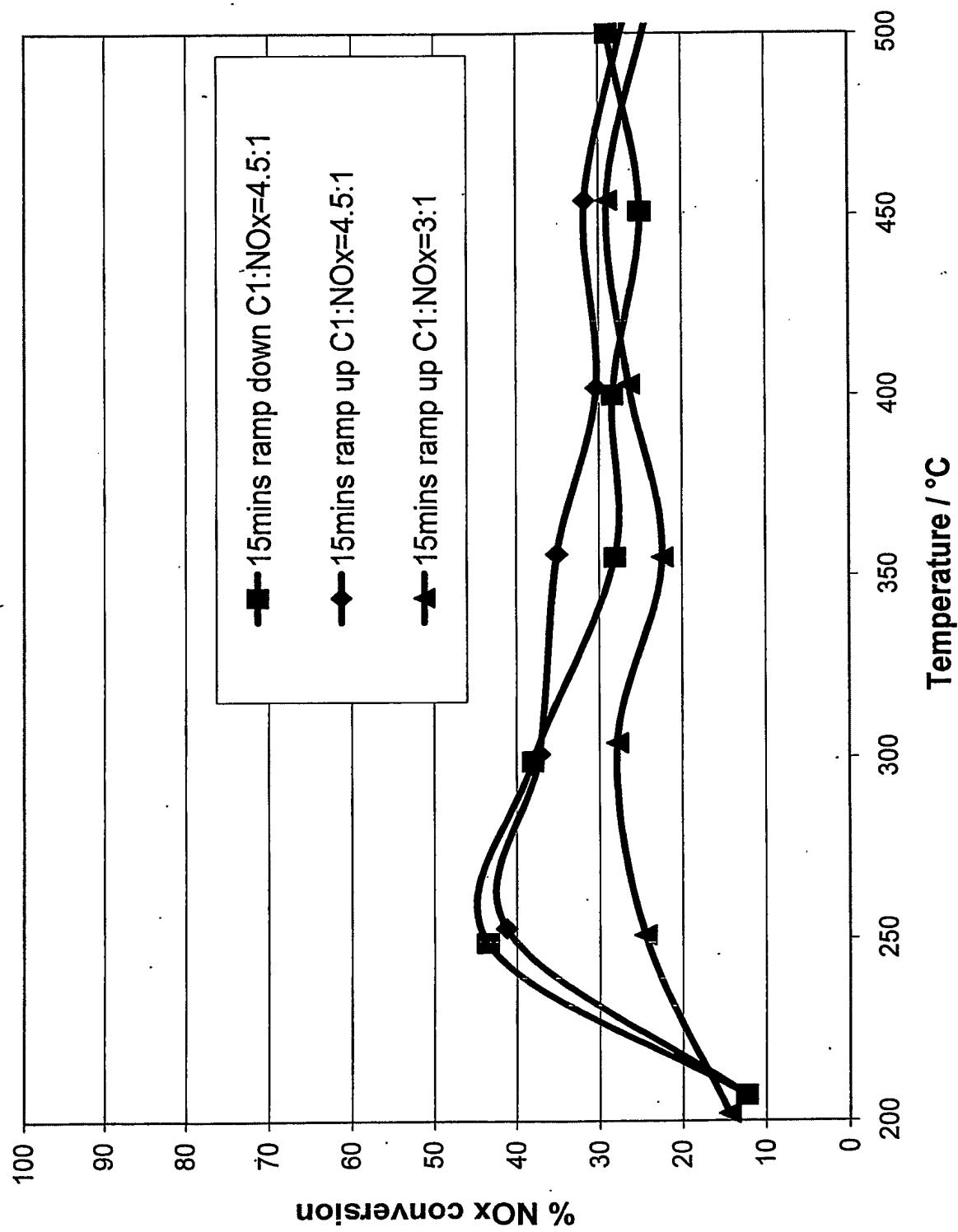




FIGURE 4



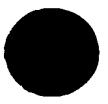


Figure 5

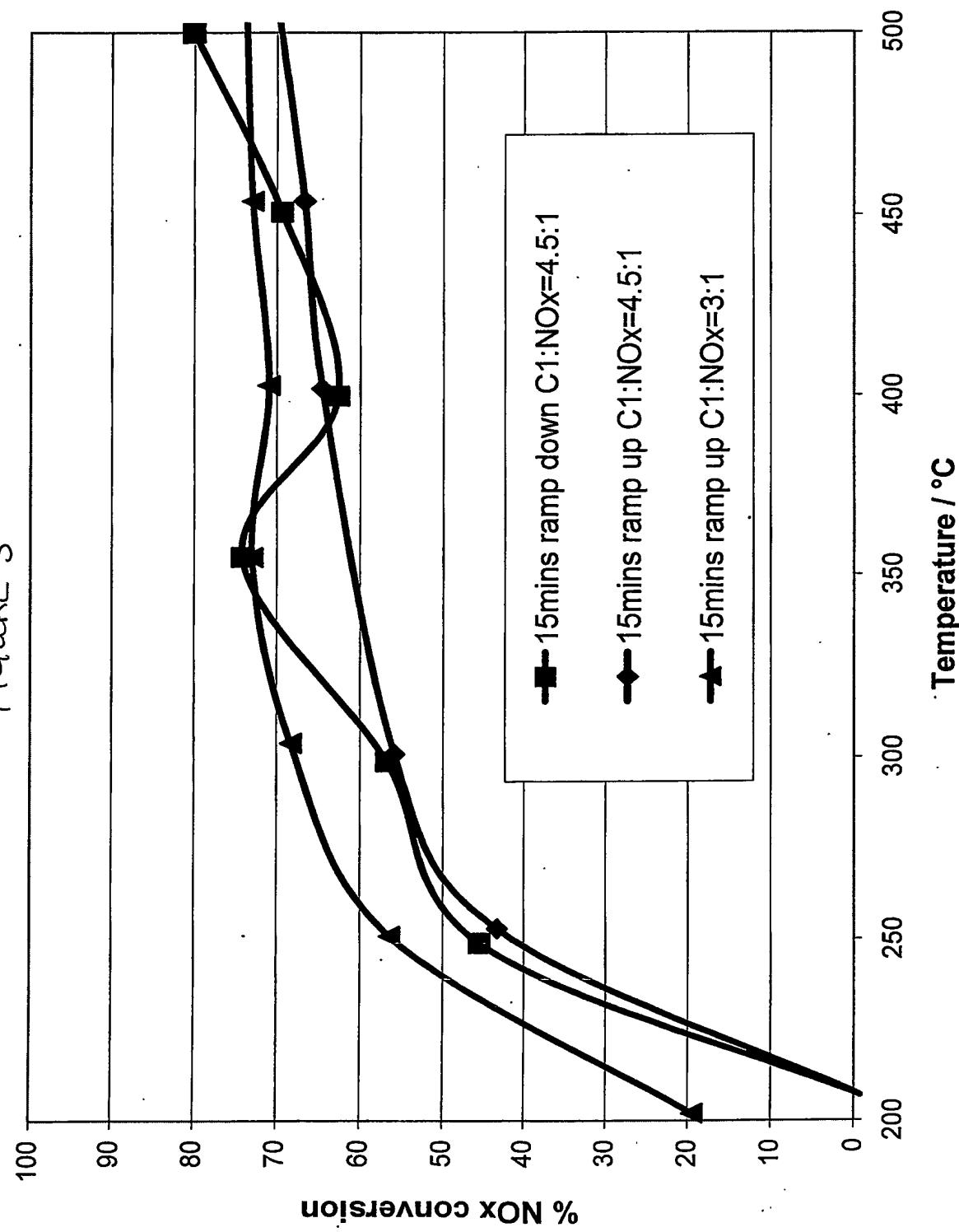
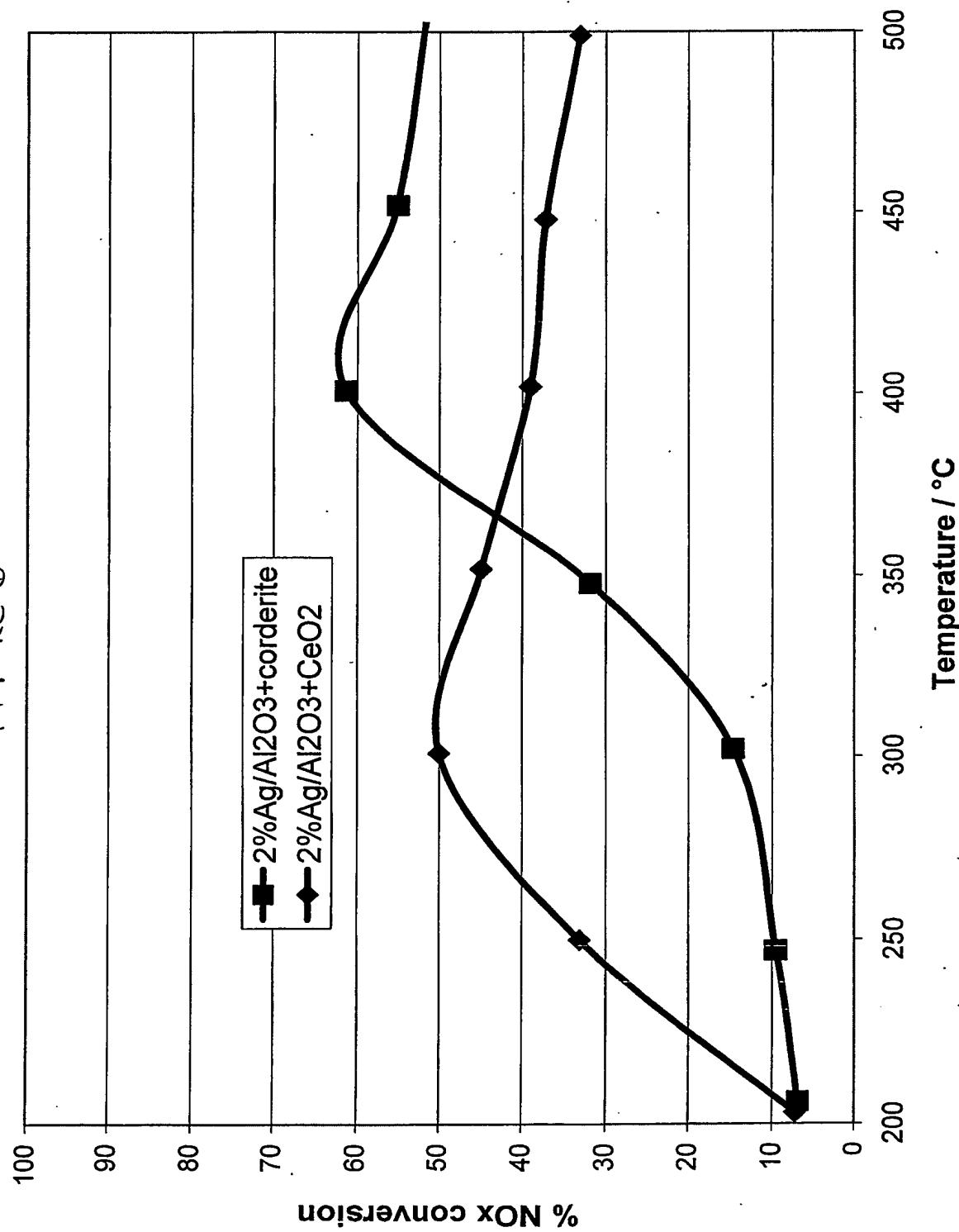




FIGURE 6



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